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REVIEW

Processing and Storage Effects on Orange Juice Aroma: A Review

Pilar Ruiz Perez-Cacho[†,‡] and Russell Rouseff*[,†]

University of Florida, Institute of Food and Agricultural Sciences, Citrus Research and Education Center, 700 Experiment Station Road, Lake Alfred, Florida 33850, and IFAPA, Centro Alameda del Obispo, Av. Menéndez Pidal s/n, 14080-Cordoba, Spain

Freshly squeezed orange juice aroma is due to a complex mixture of volatile compounds as it lacks a specific character impact compound. Fresh hand-extracted juice is unstable, and thermal processing is required to reduce enzyme and microbial activity. Heating protocols range from the lightly heated not from concentrate, NFC, to the twice heated, reconstituted from concentrate, RFC, juices. Thermal processing profoundly effects aroma composition. Aroma volatiles are further altered by subsequent time−temperature storage conditions. Heating reduces levels of reactive aroma impact compounds such as neral and geranial, and creates off-flavors or their precursors from Maillard, Strecker, and acid catalyzed hydration reactions. Off-flavors such as 4-vinylguaiacol, *p*-cymene, and carvone are the products of chemical reactions. Other off-flavors such as butane-2,3-dione, guaiacol, and 2,6-dichlorophenol are indicators of microbial contaminations. Since most orange juice consumed worldwide is processed, the goal of this review is to summarize the widely scattered reports on orange juice aroma differences in the three major juice products and subsequent aroma changes due to packaging, storage, and microbial contamination with special emphasis on results from GC-O studies.

KEYWORDS: Citrus; fruit juices; aroma-active compounds; odor volatiles; off-flavors; gas chromatography−olfactometry; thermal degradation products; packaging

INTRODUCTION

Orange juice is the most widely consumed fruit juice in the world. However, not all orange juices taste alike. The pleasant odor of freshly squeezed orange juice is distinctly different from that of many commercial juices. There is also a wide flavor range among the various types of commercial orange juice. Differences between commercially processed juices are due to the combined effects of fruit cultivar and maturity, time−temperature conditions used to stabilize the juice, the number of times it has been heated, whether the juice has been concentrated, and if concentrated, how well the volatiles lost during concentration have been restored. In addition, storage time−temperature conditions and container type can have a profound impact on juice flavor at the time of consumption. Although alternate processes have been developed, almost all commercially produced orange juice is thermally processed because thermal processing is still the most cost-effective means to reduce microbial populations and enzyme activity. However, thermal processing will reduce concentrations of some of the original juice volatiles as well as induce a complex series of chemical reactions that can ultimately produce odors foreign to freshly expressed juice (*1*). Other orange juice processing steps can alter juice flavor. One of the major quality variables of commercial juice is the mechanical pressure used to extract the juice from the fruit. High extraction pressure (hard squeeze) will produce high juice yields. Lower extraction pressure (soft squeeze) will produce less juice, but the juice will be more similar to the flavor of hand extracted juice. Extraction conditions will determine relative levels of juice and peel components and, therefore, the final flavor of the juice. The pressure used to separate the juice from the pulp (finisher pressure) will also alter the composition of the juice. Depulping can reduce some of the volatiles associated with the pulp, thus altering its flavor (*2*). Deaeration is one of the final steps applied before pasteurization and can alter orange juice volatile composition by pulling off some of the most volatile juice components along with entrained air.

Packaging materials, storage time−temperature conditions, and microbial contamination can also profoundly alter juice flavor. Juice aroma compounds can be absorbed by polymeric packaging materials (*3*−*5*), and some taints can migrate from

* Corresponding author. Tel: 863 956 1151, ext. 1477. Fax: 863 956 4631. E-mail: rrouseff@ufl.edu.
† University of Florida.
‡ Centro Alameda del Obispo.

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the packaging materials into the juice (*6*). Storage time and temperature can also alter juice aroma profiles due to Maillard, Strecker, and acid catalyzed hydration reactions. The formation of methional from methionine is probably the most significant example of Strecker degradation in orange juice. Furthermore, microbiological contaminations can alter the characteristic aroma of orange juices or produce specific off-flavors, producing consumer complaints and product rejection (*7*).

Consumers demand juice products with characteristics similar to those of freshly squeezed juices; therefore, an understanding of the changes in volatile compounds due to processing, packaging, and storage can lead to commercial juices with better flavor quality. This study summarizes and evaluates our current knowledge of aroma active volatile changes during commercial processing and storage, and odor changes due to packaging interactions as well as off-odors produced during storage via chemical or microbial means. A compilation of odor-active compounds in processed orange juice gathered from GC-olfactometry studies is also presented. To our knowledge, this is the first comprehensive review of processed orange juice flavor.

ORANGE JUICE AROMA

Flavor volatiles in fruits and vegetables are secondary metabolites formed during normal ripening and maturation largely from fatty or amino acid precursors (*8−10*). As in many fruits, the aroma of freshly squeezed orange juice is primarily attributed to aldehydes (e.g., acetaldehyde, hexanal, octanal, decanal, neral, geranial, and Z-hex-3-enal) and esters (e.g., Ethyl butanoate and ethyl-2-methylpropanoate), in addition to smaller numbers of alcohols (e.g., linalool), ketones (1-octen-3-one), and hydrocarbon terpenes (myrcene, α-pinene, and possibly limonene) (*1, 11−18*). All these odor-active compounds impart citrus, green, sweet fruit, and floral odor notes to fresh hand-extracted juice aroma (*19−21*). Fresh from the tree, hand squeezed orange juice is the sensory standard for what orange juice should taste like. However, few people can enjoy juice in this manner as citrus can only be grown in certain regions of the world. An ideal processed orange juice would maintain all the sensory perceptions of the freshly squeezed juice. However, certain chemical laws dictate that when juices are subject to thermal processing and postproduction packaging/storage conditions, specific components will undergo reactions that will alter the sensory profile. Several of the original sensory descriptors will be lost or diminished, and new sensory attributes will eventually appear. As a result, there have been extensive searches for alternate processing techniques, which would maintain more of the fresh juice aroma attributes (*22, 23*). As it currently stands, the aroma of orange juice is profoundly affected by processing, packaging, and storage conditions. The aroma changes are largely determined by the time−temperature conditions used to stabilize the juice, whether the juice is evaporated, the type of container, and storage conditions as well as the skill and care of the manufacturer.

Juice Extraction. Botanically, oranges (*Citrus sinensis*) are berries with an aromatic peel and a fleshy interior. The peel consists of an orange colored outer layer called the flavedo, which contains oil glands and pigments, and a white spongy inner layer called the albedo. The fleshy interior or endocarp consists of wedge shaped sections (segments) filled with numerous fluid-filled sacs or vesicles. These juice sacs constitute the edible portion of a citrus fruit and provide the primary source of the citrus juice (*24*). The juice is liberated when the juice vesicles are ruptured during physical extraction. This complex chemical mixture contains many compounds previously separated within the intact fruit but are now free to interact. In addition, peel oil from the flavedo is commingled with the juice when the peel is broken during juice extraction. The juice also contains a native amount of oil, which is slightly different in composition than that from peel (flavedo). Therefore, the type of commercial extractor and extractor pressures will determine the relative levels of peel versus juice oils and the composition of the juice volatiles, and, therefore, the overall flavor of the juice. Hand extraction is always the mildest extraction condition and will usually contain the least amount of peel oil. However, it has been demonstrated that even hand extraction can introduce small amounts of peel oil components into the juice (*25, 26*). Mechanically squeezed orange juice contains higher concentration levels of certain aldehydes (octanal, nonanal, and decanal) and terpenes (mainly, limonene, myrcene, and linalool) than fresh hand-extracted juices, as they are also present in the peel oil (*27, 28*). Consequently, mechanically squeezed orange juices will have a sensory profile somewhat different from that of comparable hand squeezed juices.

Juice Finishing/Pulp Content. Immediately after extraction, commercially produced juices pass through a stainless steel screen to separate extraneous cell and segment wall material, and embryonic seeds from the juice. In this process, a screw press is employed to separate as much juice as possible from the unwanted solid material. The industrial term for this process is called finishing, and the pressure used to separate juice from this pulp is referred to as finisher pressure. Juice composition can be altered depending on the finisher pressure employed. High finishing pressures effectively squeeze the pulp so hard that the liquid portion of the pulp is added to the juice. Those solid particles that pass through the finisher screen are subsequently dispersed into smaller particles using homogenizers. At a latter stage, some juice manufacturers add juice sacs to produce a pulpy juice product as the physical appearance and mouth feel is what some consumers consider close to what they might prepare at home.

The finely suspended solids, which give juice its turbid appearance, are referred to as cloud. These suspended solids retain many juice volatiles. Juice volatiles partition between the insoluble pulp/cloud and aqueous serum. Hydrocarbons (mono and sesquiterpene) are almost exclusively (80−90%) associated with pulp, whereas oxygenated compounds (esters, alcohols, and aliphatic aldehydes) are more closely associated with the serum (*2, 29−31*). If the juice is clarified to remove this finely suspended material, an enormous amount of aroma compounds are eliminated (*32*), and the flavor of the clarified juice is altered. It has been reported that the volatile compounds associated with suspended solids (pulp and cloud) from a freshly squeezed orange juice represent ∼80% of total juice volatiles (*29*). Because pulp content and particle size can be controlled under commercial production conditions, commercially produced orange juices will have a different pulp content and physical distribution than hand squeezed juices (*33*).

Deaeration/Centrifugation. Deaeration is the process of removing/reducing entrained air from the juice, just prior to thermal treatment, and can affect juice quality (*34−37*). Entrained air can cause foaming related fill problems during the packaging step as well as accelerate vitamin C degradation during storage. Deaeration is employed only for those juices that will not undergo concentration as the concentration process removes entrained air along with water and most aroma volatiles. For single strength (nonconcentrated) juices, deaeration is typically accomplished by exposing the juice to a partial vacuum, although other processes such as centrifugation are available. Deaeration can significantly reduce the most volatile alcohols, aldehydes, and terpene hydrocarbon concentrations if not carefully controlled. Juice flavor quality will be diminished as many of these volatiles are important contributors to orange juice aroma. Even though ester and ketone concentrations were essentially unaffected by the deaeration process, one study (*34*) concluded that the major changes in orange juice volatiles were due to deaeration and not to pasteurization.

Thermal Processing. Heating is employed to destroy spoilage microorganisms as well as inactivate enzymes that destabilize the juice cloud and diminish juice quality during storage (*38*). Even though high temperature−short time procedures are employed, juice aroma is altered due to losses of original aroma impact volatiles (mainly aldehydes and esters) and the formation of new volatiles or their precursors. When orange juice is heated, a complex series of chemical reactions are initiated involving peel oil components, phenolic compounds, sugars, amino acids, lipids, ascorbic acid, and sulfur-containing components (*39*). Many of the products of these reactions include oxygen-, sulfur-, and nitrogen-containing compounds. Many of the most potent oxygen-containing aroma volatiles (aldehydes, ketones and alcohols) are produced from the peroxidation of unsaturated fatty acids. Heating increases the rate at which a range of alcohols such as α-terpineol are produced from acid-catalyzed hydration of terpenes, the degradation of cinnamic acids, giving rise to aromatic aldehydes and alcohols, the degradation of carbohydrates forming furanaldehydes, furanones, and other Maillard-type compounds, and the degradation of free amino acids yielding Strecker aldehydes. Although sulfur-containing compounds can be formed in natural products as a result of biochemical and enzymatic pathways, many important sulfur aroma compounds are produced during the thermal processing of food. The primary source of sulfur, from which sulfur-containing aroma compounds may be derived, are the sulfur amino acids, cysteine, cystine and methionine (*40*).

As conventional pasteurization treatment can negatively affect the flavor of orange juice, different heating processes have been tested on orange juices. Leizerson and Shimoni (*41*) examined the influence of ohmic heating on the quality and shelf life of orange juice compared to that of conventionally pasteurized orange juice. They concluded that ohmic-heated orange juice had higher aroma volatile concentrations than conventional pasteurization. The lower initial heat load during pulsed electric fields treatment might induce fewer chemical reactions, resulting in greater retention of initial flavor compounds. In addition, sensory experiments indicated that although assessors could distinguish between fresh and pasteurized samples and between pasteurized and ohmic-heated juices, they could not differentiate between fresh and ohmic-heated orange juice. Thus, ohmic heating produced a juice that retained most of the sensory attributes of the initial juice and still reduced microbial and enzymatic activity to required levels. Min and co-workers (*42*) obtained similar results in their study of microbial, nutritional, flavor, and sensory properties of orange juice exposed to commercial-scale pulsed electric field processing. Baxter and co-workers (*43*) subjected Navel orange juice to high pressure processing. GC-MS analysis demonstrated that the levels of 20 key aroma compounds in conventional temperature treatment and high pressure juice were similar. Pasteurization of orange juice using gamma irradiation has also been examined (*44, 45*). However, the elevated doses necessary for controlling microorganisms, lead to the formation of off-odors that rendered the product unacceptable. Some of the compounds responsible for these odor defects in irradiated orange juice were sulfur compounds such as dimethyl sulfide (cabbage-like odor), dimethyl disulfide (onion-like, cabbage-like odor), methanethiol (cabbage-like), and dimethyl trisulfide (cabbage-like odor).

Concentrated, Reconstituted, and Not from Concentrate Juices. Most orange juices are mechanically extracted and concentrated to reduce the cost of transportation and storage (*46*). It is simply more economical to ship a concentrated juice and add water at the destination rather than shipping juice with all its natural water content. The commercial processes of concentrating orange juice usually involves the removal of water at high temperature with slight vacuum for short times followed by recovery and concentration of volatile aromas and their addition back to the concentrated product (*47*). However, it has been noted that some juice manufacturers do not restore all the original volatiles, probably for economic reasons (*48*).

The concentrated juice can be frozen and sold as frozen concentrated orange juice, FCOJ, or stored and shipped in bulk to a distant distribution point where dilution, reheating, and packaging occurs. These juices are designated as reconstituted from concentrate, RFC. The aroma of many RFC juices are said to be heated or processed, differing from that of freshly squeezed oranges (*19, 21, 48*) This off-odor is observed most commonly in canned RFC juices, which have been heated twice, one during the concentration process and again after the juice has been diluted with local water and hot filled to sterilize the can and lid. These thermal treatments (high temperature/times) induce chemical changes in orange juices, which severely degrade the original fresh orange juice volatiles and produce new volatiles. It has been demonstrated that to generate this type of off-flavor under laboratory conditions (glass containers), orange juice must be heated to 96 °C for 6 s (*49*). Commercially, juices are heated in contact with stainless steel surfaces.

As the demand for higher quality orange juice has increased, studies have been undertaken to identify flavor changes produced in the preparation of various juice types and how to avoid or minimize them (*3, 42, 44, 50, 51*). Selected volatiles from pasteurized, not from concentrate, NFC, orange juices were not markedly different from those of fresh juice, whereas RFC juices had reduced levels of acetaldehyde, methyl acetate, ethyl acetate, and ethyl butanoate, and slightly elevated levels of decanal, octanal, and linalool (*52*). RFC orange juices were found to contain less of the highly volatile aroma compounds, which are thought to give juice a fresh, fruity note (*53*−*55*). A recent study reported marked differences in volatile levels between different orange juice types (*54*). Freshly extracted and commercial unpasteurized juices contained greater amounts of the more volatile aroma compounds than RFC (twice heated) juices. For example, fresh unpasteurized juice contained 11 to 53 times more acetaldehyde than the RFC juices. The unheated juices also contained a greater number of volatiles. In unheated juices, 11−12 esters, 13 aldehydes, and 25−27 terpenes were identified, whereas RFC juices contained only 4−6 esters, 7−8 aldehydes, and 18−20 terpenes, suggesting that the restoration of juice volatiles in the RFC juices was incomplete. Total terpene and alcohol peak areas were similar in all samples, but total aldehyde and ester concentrations were significantly higher in the unheated juices. Unfortunately, sugar/acid levels were not reported; therefore, the influence of fruit maturity on juice volatiles could not be evaluated. In another study, which developed from a sensory survey of orange juices, some canned RFC juices were not perceived as orange juice. These juices were described by a trained panel as tropical fruit/grapefruit, heated/caramel, and moldy, whereas fresh hand-squeezed orange juices completely lacked these sensory descriptors (*48*). The volatiles from these atypical orange juices were further examined using GC-O, GC-MS, and GC-PFPD using SPME. The off-flavor RFC juice contained 1 ester, 7 aldehydes, 12 terpenes, 8 sulfur compounds, 2 furanones, 2 phenols, and other minor aroma-active compounds. The atypical aroma was attributed to the strong contribution of sulfur volatiles. Four of the 12 most intense aroma peaks were sulfur compounds that included methanethiol, 1-*p*-menthene-8-thiol, 2-methyl-3-furanthiol, and dimethyl trisulfide.

ODOR CHANGES DUE TO PACKAGING AND STORAGE

Orange juice is packaged either as hot juice to sterilize the container and lid or as a chilled juice under aseptic conditions into a sterilized container and lid. The former process reduces many of the original aroma volatiles and induces off-flavor formation as the extended time at elevated temperatures promotes flavor degrading reactions. Volatile changes during orange juice storage have been the subject of research for over 40 years, even though the storage containers have changed from tin-coated steel cans and glass bottles to multilayer gable top and PET (polyethylene terephthalate) blow molded containers. Changes in aroma compounds during storage are due to storage time and temperature, oxygen content, light exposure, and container sorption or chemical contamination. Of all these factors, storage temperature is the most important (*3*). Early orange juice storage studies concentrated on readily observable and/or easily measured changes such as juice darkening (non enzymatic browning) and loss of ascorbic acid due to oxygen. Ascorbic acid loss was tracked during several storage studies and was directly related to oxygen content (*3, 56, 57*), but was not directly responsible for flavor changes. Increases in terpene alcohols were observed and attributed to acid-catalyzed hydrations (*58*). Later studies focused on furfural as an indicator of increased storage abuse (*57, 59, 60*). Although furfural can be an indicator of juice that has experienced elevated temperatures (as an early Maillard reaction product), its aroma threshold is rarely exceeded.

Storage Off-Flavors. Off-flavors are a major factor in consumer acceptance, and those specific for citrus have been reviewed (*38, 61*). In a study of off-flavors produced in canned orange juice stored at 35 °C for 12 weeks, three compounds (α-terpineol, 4-vinylguaiacol, and Furaneol) were found at elevated levels (*62*). Both 4-vinylguaiacol and Furaneol were reported in orange juice for the first time. When these three components were added to fresh juice, they produced the characteristic odor of aged or heat-abused juice. Today, we recognize that α-terpineol and Furaneol exist at low levels in pasteurized juice. It is also worth noting that the taste panel observed an increased grapefruit character in these juices that was not associated with nootkatone. This grapefruit character has recently been shown to be due to thermally induced sulfur volatiles (*48*). Of the three storage off-flavors, 4-vinylguaiacol was the most potent, and it has been extensively studied (*63*−*66*). Elevated and accelerated temperature storage studies have shown that this compound has a relatively high energy of activation requirement and its presence as an aroma active compound indicates that the juice has been thermally abused. 4-Vinylguaiacol concentrations remain essentially unchanged and never exceed its aroma threshold when stored at 4 °C for up to 16 weeks, but when stored at 40 °C, its concentration increases rapidly and exceeds its aroma threshold after only 6 weeks (*65*). α-Terpineol was reported to produce a stale, musty, or piney off-flavor when added to orange juice (*62*); however, GCO studies (*1, 67*) have demonstrated that this compound is rarely aroma active in commercial orange juices, suggesting that the added α-terpineol may have contained aroma active impurities. The odor threshold for α-terpineol in orange juice is very high, 16.6 mg/L (*68*), and rarely exceeded in commercial orange juices.

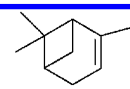
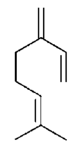
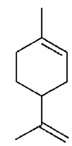
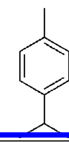
Storage Temperature. There is a general agreement in the literature that storage temperature is the major factor limiting shelf life in juices (*3, 4, 69*−*71*). The overall aroma of orange juices does not change significantly if they are stored at refrigerated (4−6 °C) temperatures for up to 16 weeks, but changes in aroma compounds have been observed in orange juices stored at higher storage temperatures. A gradual decrease in several flavor components (ethyl butanoate, hexanal, octanal, neral, and geranial) combined with an increase in undesirable compounds (ethyl acetate, α-terpineol, and furfural) in aseptically package orange juice was observed during 8 months of storage at 21 and 26 °C (*72*). A linear increase in α-terpineol with increasing storage time due to limonene degradation in a nonoxidative pathway was observed (*69*). In another storage study comparing concentration differences due to storage temperature, commercial juices stored for 12 weeks at −18 °C contained approximately 1 ppm of α-terpineol, whereas the same canned juices stored at 35 °C for the same time period reached concentrations ranging from 3.4 to 5.5 μg/mL (*62*). Since α-terpineol is formed more rapidly from linalool than limonene, the linalool/α-terpineol ratio has been suggested as a means of evaluating orange juice storage time/conditions (*73*).

Packaging Interactions. Several studies have shown that considerable amounts of aroma compounds can be absorbed by the food-packaging materials (*4, 5, 72*). Some taints could migrate from the packaging material into the juice (*74*). In a recent flavor absorption study involving low-density polyethylene (LDPE), polycarbonate (PC), and polyethylene terephthalate (PET) in contact with orange juice, it has been shown that no sensory significant differences were found between polymer-treated samples and controls (*4*). Thus, in spite of the fact there are losses of flavor volatiles, they do not influence orange juice flavor perception significantly after 29 days of dark storage at 20 °C. This may be due to the fact that the major components sorbed by the packaging material were terpenes with little odor activities. This finding agrees with a similar GC-O study by Marin and co-workers, who concluded that the plastic polymers LDPE and Surlyn did not significantly alter the odor-active volatiles in orange juice, even though a significant decrease in limonene was observed (*75*). Several volatiles, including ethyl acetate, were found to increase in an aseptically packaged fruity soft drink during storage (*74*). This increase was due to the migration of solvent from laminated containers, and it varied widely between individual packages. Most packaging studies examined oxygen barrier properties of various materials under various storage conditions (*3, 76, 77*), whose effect will be discussed in the next section. Storage temperature was more important than oxygen barrier properties.

Oxygen Effects. Although considerable efforts are made to minimize oxygen content in the production of citrus juices, there is little evidence that the amount of oxygen directly alters the aroma of citrus juices during storage. In a five month (22 °C) single strength juice storage study with dissolved oxygen levels of 0.6, 1.8, 6.5, and 10.1 ppm stored, oxygen removal did not improve product shelf life based on sensory evaluations (*56*). However, it is quite likely that under these conditions other flavor degrading reactions may have masked any effect due to oxygen. A later study (*78*) examined the effect of enzymatic deoxygenation, using glucose oxidase-catalase immediately after juice extraction but found no improvement in the shelf life of pasteurized juices due to oxygen reduction. The primary effect of oxygen is to oxidize ascorbic acid producing dehydroascorbic acid (*79*), which contains an α-dicarbonyl group and can take part in the production of Strecker aldehydes from the corresponding amino acids. The production of methional from methionine is probably due to this process.

Light Effects. The combination of light and oxygen are necessary factors in lipid peroxidation, producing a number of potent aldehydes (*80*). Both factors have been examined as factors in flavor changes observed during orange juice storage (*81*−*83*) but not attributed to lipid peroxidation. When orange juice is exposed to light in the presence of oxygen, an oxidized or cooked off-flavor was observed (*81*). Off-flavors were not observed if the samples were exposed to light but oxygen was excluded (*83*). Interestingly, it had been demonstrated in lipid storage studies that free fatty acids increased 3-fold during a 16 month storage study at 4 °C and 8-fold at 29.4 °C (*84*). It has yet to be established that the orange juice off-flavors observed in the presence of light and oxygen were due to lipid peroxidation.

Table 1. Aroma Active Hydrocarbon Terpenes in Processed Orange Juice

α-Pinene (2-Pinene)		Resin, pine tree,	(*67, 95, 97*)
Myrcene (2-Methyl-6-methylene-2,7-octadiene)		Mossy, geranium, green, metallic	(*1, 48, 67, 97*)
Limonene (*p*-Mentha-1,8-diene)		Citrus-like, minty	(*1, 67, 75, 95, 97*
p-cymene		minty, solventy	(*48*)

ODOR CHANGES DUE TO MICROBIAL CONTAMINATION

Microorganisms (molds, yeasts, and bacteria) can cause spoilage and sensory defects in fruit juices. Lactic acid bacteria are found most frequently in fruit juices, and orange juice spoilage is usually characterized by a vinegar or butter-milk odor (*85*). The metabolic products of this biological contamination include diacetyl (butane-2,3-dione), acetoin (3-hydroxy-2-butanone), and odor active acids (e.g., acetic acid and butyric acid). Recently, different strains of *Alicyclobacillus* have been associated with medicinal or antiseptic off-flavors in orange juice (*7*). The medicinal off-flavors associated with *Alicyclobacillus* species have been attributed to metabolite products of these bacteria and have been identified as guaiacol (2-methoxyphenol) (*86*) along with two halogenated compounds (2,6-dichlorophenol and 2,6-dibromophenol) (*87*). The two halogenated phenolic compounds are not produced by all species and strains of *Alicyclobacillus* (*7*), but guaiacol is always the primary metabolic product.

ODOR-ACTIVE VOLATILES IN PROCESSED ORANGE JUICES

Early orange juice studies focused on identifying and quantifying volatiles found at the highest concentrations (*51, 52, 88*−*92*). At the time of these studies, it was thought that those volatiles found at the highest concentration should contribute the most to flavor. Even before the advent of gas chromatography−olfactometry, it had been shown that minor components make major contributions to flavor (*93*), and since then, it has been estimated that less that 5% of all food volatiles possess aroma activity (*94*). Gas-chromatography−olfactometry (GC-O) has been shown to be a powerful technique to characterize and tentatively identify odor-active compounds, which are typically confirmed by MS whenever possible. Recent orange juice studies (*67, 75, 95*−*98*) have employed GC-O to determine which volatiles are responsible for the sensory changes in orange odor due to thermal processing and storage and are described in the following sections.

Hydrocarbon Terpenes. Hydrocarbon terpenes are the predominant chemical class within orange volatiles and comprise over 95% of the peel oil. However, their contribution to odor perception is limited because of high odor thresholds. Limonene, myrcene, α-pinene, and *p*-cymene are the four terpene hydrocarbons commonly reported in processed orange juices (**Table 1**). Limonene is the most abundant terpene hydrocarbon in orange juice, and its concentration in processed orange juice is much higher than in fresh hand squeezed juices. This variability arises because most of the limonene comes from peel oil and is introduced into the juice during mechanical extraction. In spite of its high concentration, limonene is not a key flavor impact compound in orange juice. Nevertheless, limonene is a necessary component of any orange juice odor model, although its exact function is still uncertain (*99*). Moreover, many reports have mentioned that the high concentration of this terpene is associated with off-odors (*39, 73*) or a negative sensory mouth feel commonly referred to as peel burn. Thus, limonene, because of its unsaturated sites, can also thermally induce products such as carvone via an oxidative pathway (*100*), which can degrade the flavor quality of the juice. Myrcene is the next most abundant terpene. It has a green, mossy odor and is a much stronger odorant than limonene, even though limonene is present in considerably higher concentrations. α-Pinene with a pine-tree, resin aroma can make a positive background contribution to orange aroma, although its level in orange juice directly depends on the peel oil content of the juice.

Oxygen-Containing Volatiles. Oxygenated aroma compounds make a major contribution to the aroma of numerous foods. In fruits and vegetables, they can be formed from unsaturated terpenes, fatty acids, sugars, or amino acid precursors.

Aldehydes. Aldehydes are secondary metabolites formed during normal ripening and maturation of orange fruits. They are important in terms of orange odor quality and their concentrations increase with fruit maturity (*91*). Some aldehydes impart a pleasant green and citrus note to fresh squeezed orange juices, whereas others impart fatty, metallic notes. Before individual aldehydes could be quantified, chemical tests for total aldehydes were used as a rough measure of flavor quality of citrus oils (*101, 102*). Some reactive aldehydes such as acetaldehyde, (*Z*)-hex-3-enal, neral, and geranial are present at super threshold levels in fresh squeezed juice but at diminished levels in processed orange juices if at all. Much of the perceived flavor quality of orange juice is produced by the relative amounts of these aldehydes.

Twelve aroma-active aldehydes have been identified in processed orange juices (**Table 2**). Neral and geranial (formerly called citral) are important volatile components of orange juice and peel oil. These two isomeric monoterpene aldehydes are typically found in the ratio of 2:3 and possess a lemon, citrus-like odor. They are highly unstable, and their concentrations are diminished during thermal processing and storage. α-Sinensal and β-sinensal are two isomeric forms found as a 2:1 ratio of β-sinensal to α-sinensal and are described with a sweet aroma.

Long-chained unsaturated fatty acids are important precursors of many volatile off-odors compounds, such as alk-2,4-dienals, alk-2,6-dienals, and aliphatic saturated aldehydes (e.g., hexanal, octanal, and decanal). It has been shown that concentrations of all of these aldehydes increase when orange juice is heated (*1, 14, 97*). Concentrations of several straight-chain aldehydes such as octanal, nonanal, and decanal are found at higher levels in processed juices because they are constituents of the co-mingled peel oil introduced during juice extraction (*103*). Most of the alk-2,4-dienals and alk-2,6-dienals possess a fatty, metallic, or fried odor that could contribute to off-flavors when out of balance with the more desirable aldehydes. As seen in **Table 2**, there are nine aliphatic aldehydes, which have been identified in processed juices using GC-Olfactometry.

Some important aldehydes are restored in reconstituted from concentrate, RFC, juices by the addition of aqueous essence and peel oil after the original volatiles were lost during the concentration process. However, because of the relatively low economic value of this product, it is rare that all of the aroma-active aldehydes are restored. The incomplete or inadequate restoration of these volatiles contributes to the lower flavor quality perception of these juices compared to that of fresh squeezed or not from concentrate, NFC, juices.

Esters. Esters also make an important contribution to orange odor, and the level of total esters in aqueous essence has been used as a quality index (*16*). However, when fruits are homogenized such as in the processing of juice, many esters are rapidly hydrolyzed by hydrolase enzymes, thus reducing their concentrations. Also, their concentrations are dramatically reduced after thermal processing. Thus, only 4 esters have been reported in processed juices using GC-O (**Table 2**). Ethyl butanoate is one of the most potent odorant of processed juices, and it is an important contributor to desirable flavor in orange products (*67, 97*). Lower levels of ethyl butanoate, as well as total esters, have been observed in processed juices (*52*) compared to the initial juice. Esters are responsible for the fruity top-notes present in fresh juice that are often missing or diminished in thermally processed orange juice products.

Wine lactone (3a,4,5,7a-tetrahydro-3,6-dimethyl-2(3*H*)-benzofuranone) is a cyclic ester (lactone), which has one of the highest flavor dilution (FD) factors in processed orange juice (*67*). It may be speculated that wine lactone is formed from linalool via a hydrolytic conversion (*104*). However, it should be noted that wine lactone has never been reported as aroma active in any orange juice head space study. It has only been observed in solvent-extracted samples, suggesting that this potent compound (along with vanillin) has a very low vapor pressure at room temperature and at 37 °C.

Alcohols. Five terpene alcohols with odor activity have been reported in processed orange juices using GC-O (**Table 2**). Linalool is the most aroma intensive alcohol and possesses a distinctive floral, sweet odor. Although linalool is also present in fresh hand squeezed juices, its concentration level is higher in processed oranges juices as most of it originates from the peel oil (*26*). Highest levels of linalool are observed in RFC juices as a result of flavor restoration efforts. Excessively high levels of linalool and/or others terpenes can produce flavor imbalances.

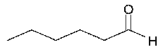
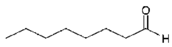
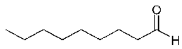
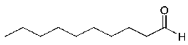
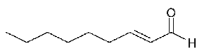
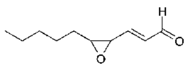
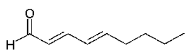
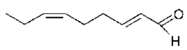
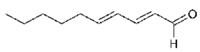
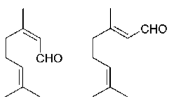
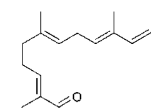
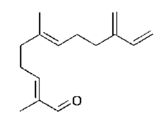
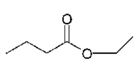
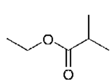
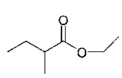
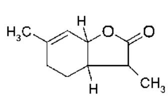
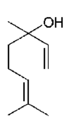
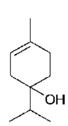
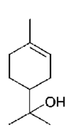
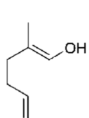
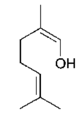
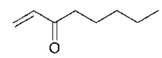
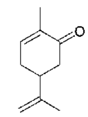
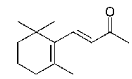
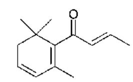
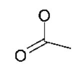
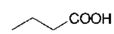
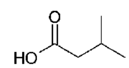
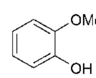
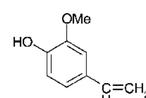
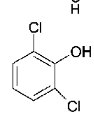
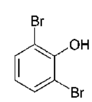
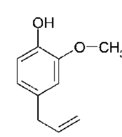
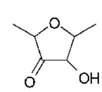
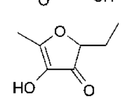
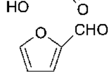
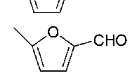
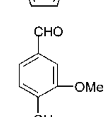
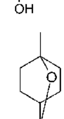
Under orange juice acidic conditions (pH ~3.8), hydrocarbon terpenes undergo a series of oxidative hydration−dehydratation reactions that produce alcohols (e.g., terpinen-4-ol, α- terpineol, and β-terpineol) and other products (e.g., *p*-cymene or terpinolene) (*38, 61*). The formation of these thermally induced terpene alcohols can alter the overall sensory properties of orange juice. Linalool degrades primarily to α-terpineol but also to 1,8-cineole, geraniol, nerol, and terpinen-4-ol (*26, 73, 105*). Although both limonene and linalool can undergo acid-catalyzed hydrations to form α-terpineol and juices contain much less linalool relative to limonene, linalool is more reactive and produces most of the formed α-terpineol. Although Tatum and co-workers reported that orange juices spiked with 2.5 ppm of α-terpineol imparted a stale, musty, or piney note into the juice (*62*), GCO studies have shown that α-terpineol is rarely aroma active. α-Terpineol is generally considered a marker for heat-abused orange juices (*66, 105*) rather than something that directly impacts juice flavor. Bazemore and co-workers reported that the odor of terpinen-4-ol, described as metallic/stale, was judged stronger in the heat-abused juice than in the lightly heated juice. A recent study of the off-odors in canned RFC orange juices observed elevated levels of *p*-cymene, 1,8-cineole, geraniol, nerol, terpinen-4-ol, and 4-vinylguaiacol (*48*).

Ketones and Acids. One of the most intense odor-active aliphatic ketones in orange juice is 1-octen-3-one. It is a lipid decomposition product, and its odor has been described as metallic and mushroom-like. It is extremely potent with an odor threshold in water of 0.005 μg/L (*10*). Two ketone norisoprenoids, β-ionone and β-damascenone, are also odor-active compounds in processed orange juices and possess a floral note at their juice concentrations. Nevertheless, they have only been reported by a single research group (*48*). β-Ionone has been reported as among the more odor-active volatiles in freshly squeezed Valencia orange juices (*11*). Its odor is described as sweet floral and raspberry in orange juice and has an extremely low odor threshold in both water and orange juice pump-out (*68, 106*). β-Damascenone is described as tobacco, apple, and floral and contributed to 19% of the floral compounds in freshly squeezed orange juice analyzed by GC-O (*13*). According to these authors, its concentration ranged from 0.122 to 0.281 μg/L in orange juice not from concentrate and as high as 0.145 to 0.690 μg/L in reconstituted orange juice from concentrate (*107*). This norisoprenoid can be produced from neoxanthin (*108*).

Carvone is an off-flavor ketone produced from the oxidation of limonene and can be found in two enantiomeric forms: *S*-(+)-carvone, which smells like caraway, and its mirror image, *R*-(−)-carvone, which smells like spearmint. It is thought to degrade orange juice quality, and its concentration is expected to increase as a result of thermal treatment and oxidative storage (*1, 48, 67*). In previous orange juice studies, carvone odor was described both as a caraway-like odor quality (*67*) and also as a minty odor (*48*).

A few acids have been identified as odor-active compounds in processed juice, although their contribution to the overall juice odor is minimal (*48, 67, 95*). They are thought to be formed by fatty acid oxidation or by microbiological spoilage.

Table 2. Oxygen Containing Compounds in Processed Orange Juice

Odor-active compound	Chemical structure	Odor quality	Reference
Aldehydes			
Hexanal		Green, grassy, soapy	(48, 67)
Octanal		Green, citrus, orange peel	(48, 67, 95, 97)
Nonanal		Citrus-like, soapy	(48)
Decanal		Green, citrus-like, fatty	(1, 48, 67, 97)
(E)-2-Nonenal		Fatty, tallowy, green, metallic	(1, 48, 67, 97)
(E)-4,5-Epoxi-(E)-dec-2-enal		Metallic, fatty	(48, 67)
(E, E)-Nona-2,4-dienal		Fatty, waxy, soapy	(67)
(E, Z)-Nona-2,6-dienal		Cucumber-like, green	(97)
(E, E)-Deca-2,4-dienal		Fried, fatty, wax	(48)
Citral (neral + geranial) (Z)- 3,7-dimethyl-2,6-octadienal + (E)-3,7-dimethyl-2,6-octadienal		Citrus-like, lemon	(75)
α-Sinensal (2,6,10-trimethyl-2,6,9,11-dodecatetraenal)		Sweet floral	(48)
β-Sinensal (2,6-dimethyl-10-methylene-2,6,11-dodecatrienal)		Sweet citrus, aquarium	(48)
Esters			
Ethyl butanoate		Fruity, sweet	(1, 48, 67, 75, 95, 97)
Ethyl 2-methylpropanoate		Fruity	(67, 97)
Ethyl-2-methylbutanoate		Fruity	(67, 75)
Winelactone (3a,4,5,7a-tetrahydro-3,6-dimethyl-2(3H)-benzofuranone)		Sweet, spicy	(48, 67)
Alcohols			
Linalool (3,7-dimethyl-1,6-octadien-3-ol)		Floral, sweet	(48, 67, 75, 95, 97)
Terpinen-4-ol (*p*-Mentha-4-en-1-ol)		Metallic, solventy	(1, 48, 95, 97)
α-Terpineol (*p*-Menth-1-en-8-ol)		Sweet floral, terpene-like	(95, 97)
Geraniol (E)-3,7-dimethyl-2,6-octadien-1-ol)		Floral, green	(48)
Nerol (Z)-3,7-dimethyl-2,6-octadien-3-ol)		Sweet fruit, cashew	(48)
Ketones			
1-Octen -3-one		Mushroom-like	(48, 67, 97)
Carvone (*p*-Mentha-6,8-dien-2-one)		Minty, caraway	(48, 67, 95)
β–Ionone (E)-4-(2,6,6-trimethyl-cyclohex-1-enyl)-but-3-en-2-one)		Floral, raspberry	(48)
β-Damascenone (2E)-2,6,6-trimethyl-1,3-cyclohexadienyl-2-buten-1-one)		Honey, sweet floral	(48)
Acids			
Acetic acid		Sour, pungent	(48, 67, 95)
Butanoic acid		Sweaty, rancid,burnt	(67, 95)
2/3-Methylbutanoic acid		Sweat, rancid	(67)
Phenols			
Guaiacol (2-Methoxy-phenol)		Medicine, disinfectant	(7, 48)
4-Vinylguaiacol (2-methoxy-4-vinylphenol)		Spicy, musty	(1, 48, 97)
2,6-Dichlorophenol		Medicine	(7)
2,6-Dibromophenol		Medicine	(7)
Eugenol (2-methoxy-4-(2-propenyl) phenol)		Clove-like	(48)
Furans and Furanones			
Furaneol (4-hydroxy-2,5-dimethyl-3(2H)-furanone)		Burnt sugar, caramel	(48)
Homofuraneol (2-ethyl-4-hydroxy-5-methyl-3(2H)-furanone)		Caramel	(48)
Furfural (2-furancarboxaldehyde)		Burnt	(95)
5-Methylfurfural (5-methyl-2-furancarboxaldehyde)		Sweet, citrus	(95)
Others			
Vanillin (4-hydroxy-3-methoxy-benzaldehyde)		Vanilla-like, sweet	(48, 67, 75)
1,8-Cineole (1,3,3-trimethyl-2-oxabicyclo(2.2.2)octane)		Citrus-like, minty	(1)

Phenols. As seen in **Table 2**, five odor-active phenols have been reported in processed orange juices, which individually and collectively reduce flavor quality. The potent off-odor, 4-vinylguaiacol, is perhaps the most important. It is produced from an odorless precursor, ferulic acid, which is capable of producing both vanillin and 4-vinylguaiacol. Most ferulic acid in orange juice is found in bound forms such as glycosides, esters, and amides. However, acidity, thermal processing, and subsequent high-temperature storage provide ideal conditions for the release of ferulic acid from its bound forms (*63, 109*). Ferulic acid can also be converted to 4-vinylguaiacol by yeast, which has been found in concentrated orange juice (*110*). The evidence for the formation of 4-vinylguaiacol from ferulic acid has been demonstrated in a model juice system. In terms of relative amounts, the majority of ferulic and other cinnamic acids are found in the peel. Increased mechanical pressure during fruit extraction will increase juice yield but may affect product flavor due to the introduction of peel components into the juice. Many of these peel-derived phenolics are off-odor precursors.

Vanillin is an odor-active phenolic found routinely in solvent-extracted fresh orange juice samples but is not one of the most potent odorants. At low concentrations, it might positively contribute to overall juice aroma, but its concentration is significantly higher in thermally processed juice (*67*) because of the thermal degradation of ferulic acid and at that point is typically a flavor defect.

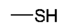
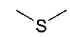
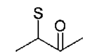
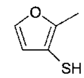
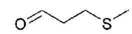
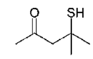
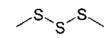
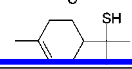
Three additional aroma active phenols have been reported in processed orange juices: guaiacol, 2,6-dichlorophenol, and 2,6-dibromophenol. These compounds are responsible for the medicinal or antiseptic off-flavors in thermally processed orange juices contaminated by different species and strains of *Alicyclobacillus* (*7*) and have been previously discussed.

Furans and Furanones. Thermal degradation of sugars, amino acids, and ascorbic acid can produce off-flavors and nonenzymic browning products (*111*). These heat-induced degradations produce acids (acetic or butyric acid), furans (e.g., furfural and 5-methyl furfural), furanones (e.g., 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone and 2-ethyl-4-hydroxy-5-methyl-3(2*H*)-furanone), ketones, cyclopentanones, pyranones, and pyrroles (*38, 62, 112*). Some of them impart sweet, caramel-like, or burnt sugar-like sensory impressions. However, other reaction products such as furfural and 5-methylfurfural, whose concentration increases due to thermal processing (*95*), are rarely aroma active as they have high odor thresholds. Because furfural is formed by oxidative degradation of ascorbic acid (*39*), it has been used as an indicator of thermal abuse.

Furanones are Maillard reaction products, which have also been identified in heated orange juices (*62*). Furaneol and homofuraneol have been identified as odor-active compounds in canned orange juices (*48*). They were potent odorants in these juices, inducing caramel odor qualities. The canned juices were described with a cooked/caramel odor defect, and these two Maillard products contributed to this overall quality degrading sensory impression in the juice.

Sulfur and Nitrogen-Containing Volatiles. Sulfur compounds are potent aroma volatiles in many foods. Although they are present at extremely low concentration levels, they are often significant in terms of odor activity as they often have even lower odor thresholds. Because they are typically found at levels close to or below the detection limits of many instruments, they are difficult to measure and have often been overlooked (*113, 114*). Although they can be formed in natural products by enzymatic and/or biochemical pathways, many important sulfur compounds are also thermally generated (*114*).

Table 3. Sulfur Containing Volatiles Found in Processed Orange Juice

Odor-active compound	Chemical structure	Odor quality	Odor thresholds in water as ug/kg
Methanethiol (*48*)		Cabbage-like,	0.2 (*121*)
Dimethyl silfide (*48*)		Sulfur, moldy	0.3 (*122*)
3-mercapto-2-butanone (*48*)		Sulfur, burning-tire	
2-methyl-3-furanthiol (*48, 96*)		Meaty, vitamin B	0.007 (*123*)
Methional (3-methylthio-propanal) (*48, 67, 96, 97*)		Cooked potato	0.2 (*124*)
4-mercapto-4-methyl-2-pentanone (*48*)		Sulfur, tropical	1 x 10^{-4} (*125*)
Dimethyl trisulfide (*48*)		Cabbage-like	0.01 (*126*)
1-*p*-Menth-1-ene-8-thiol (*48, 67, 97*)		Grapefruit-like, passion fruit	2 x 10^{-5} (*127*)

Volatile sulfur compounds are also important as potential off-flavors in heated citrus products. However, there have been few reports primarily because of the difficulty in measuring them. Earlier studies reported finding traces of sulfur volatiles such as hydrogen sulfide, methanethiol, dimethyl sulfide, dimethyl disulfide, sulfur dioxide, and carbonyl sulfide in the headspace of different citrus juices (*58, 115*−*117*). Recent works have reported finding 3-(methylthio)-propanal (methional), 2-methyl-3-furanthiol, and 1-*p*-menthene-8-thiol in both fresh (*13, 14, 96*) and processed orange juices (*67, 96*) as odor-active compounds (**Table 3**). Moreover, eight sulfur volatiles with odor activity (methanethiol, dimethyl sulfide, 3-mecapto-2-butanone, 2-methyl-3-furanthiol, methional, 4-mercapto-4-methyl-2-pentanone, dimethyl trisulfide, and 1-*p*-menthene-8-thiol) have been identified as off-odors in RFC canned orange juices (*48*). As canned RFC juices are heated twice, it is possible that the majority of sulfur compounds identified were derived from thermal processes and increased by ambient temperature storage. The prominent tropical fruit/grapefruit-like sensory attribute that these juices exhibited were correlated with such major grapefruit juice aroma impact compounds such as 1-*p*-menthene-8-thiol and 4-mercapto-4-methyl-2-pentanone. Elevated levels of 1-*p*-menthene-8-thiol were also observed in grapefruit juices that had been heated (*118*). Furthermore, Tatum and co-workers (*62*) reported a grapefruit-like aroma in canned orange juices that had been thermally abused, although they could not identify the aroma-active compound responsible for this odor. All these sulfur-containing aroma compounds are primarily derived from thermally induced reactions involving sulfur-containing amino acids, such as methionine. Thus, methional can be formed from methionine by Strecker degradation (*96*), whereas 2-methyl-3-furanthiol can be produced either in the Maillard reaction (*119*) or from the degradation of thiamin (*98*). 3-Mercapto-2-butanone may be derived from the Maillard reaction as well (*119*), and methanethiol oxidizes easily to dimethyl disulfide, which can disproportionate to dimethyl sulfide and dimethyl trisulfide (*9*). Sulfur volatiles tend to be flavor degrading volatiles when present above threshold levels.

Finally, a single nitrogen-containing compound, 2-isopropyl-3-methoxy-pyrazine, has been identified in processed juice as an odor-active volatile (*67*). This pyrazine is a powerful odorant that is a metabolic byproduct in some plant-based foods but is also produced by certain microorganisms. Recent studies have reported that *Streptomyces griseus* in tainted apple juice was responsible for these pyrazines (*120*).

ABBREVIATIONS USED

GC-O, gas chromatography−olfactometry; NFC, not from concentrate; RFC, reconstituted from concentrate; FCOJ, frozen concentrated orange juice.

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